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SPECIFICATION FOR 3-NITRO-4-AMINOTOLUENE

(First Revision)

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Gr 1 *March* 1987

AMENDMENT NO. 1 MARCH 2002
TO
IS 7364 : 1986 SPECIFICATION FOR 3-NITRO-4-AMINOTOLUENE

(First Revision)

(*Page* 3, *clause* 0.2, *Structural formula*) — Insert the following below the structural formula:

'(CAS No. 89-62-3)'

[*Page* 4, *Table* 1, *Sl No.* (iv)] — Insert the following after Sl No. (iv):

(1)	(2)	(3)	(4)	(5)
v)	Matter insoluble in methanol, percent by mass, *Max*	0.2	when tested as per IS 5299 : 2001'	—

(PCD 11)

Reprography Unit, BIS, New Delhi, India

IS : 7364 - 1986

Indian Standard

SPECIFICATION FOR 3-NITRO-4-AMINOTOLUENE

(First Revision)

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Indian Standard

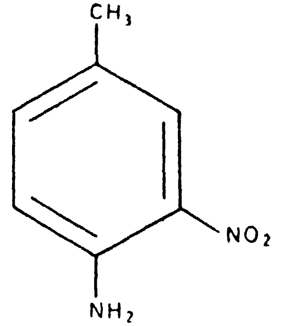
SPECIFICATION FOR 3-NITRO-4-AMINOTOLUENE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 24 March 1986, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 3-Nitro-4-aminotoluene ($C_7H_8O_2N_2$) is an intermediate used in the manufacture of pigments. It is also used as a diazo component in azoic dyeing, when it is called fast red GL base. It is also known as *m*-nitro-*p*-toluidine, 3-nitro-4-toluidine, 4-methyl-2-nitroaniline and 3-nitro-*p*-toluidine. It is represented by the following structural formula:



3-NITRO-4-AMINOTOLUENE
(Molecular Mass 152)

0.3 This standard was first published in 1974. The Committee responsible for the preparation of this standard decided to revise it mainly to stipulate the maximum limit of impurities such as 4-toluidine and 2-nitro-4-toluidine. The requirement of crystallizing point and assay have also been revised. Besides, thin layer chromatographic test method has been introduced for assay and estimation of impurities.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 3-nitro-4-aminotoluene.

2. REQUIREMENTS

2.1 Description — 3-Nitro-4-aminotoluene shall be reddish orange crystalline powder insoluble in water and soluble in concentrated sulphuric acid and common organic solvents.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 3-NITRO-4-AMINOTOLUENE

SL No.	CHARACTERISTIC	REQUIREMENTS	METHOD OF TEST, REF TO	
			IS : 5299-1969*	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Crystallizing point, °C, *Min*	114	7	—
ii)	Assay, percent by mass, *Min*	98·5	12†	—
iii)	4-Toluidine, percent by mass, *Max*	0·5	—	A
iv)	2-Nitro-4-toluidine, percent by mass, *Max*	0·5	—	

*Methods of sampling and tests for dye intermediates.

†Dimethyl formamide (15 ml for 1 g of sample) shall be used as solvent.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979†) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information.

a) Name of the material;

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*Rules for rounding off numerical values (*revised*).

†Specification for steel drums (galvanized and ungalvanized) (*second revision*).

- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number; and
- d) Tare, net and gross mass.

3.2.1 Each container shall, in addition, bear the minimum coutionary notice worded as under:

'DANGER ! HAZARDOUS SOLID AND VAPOUR RAPIDLY ABSORBED THROUGH SKIN'

3.2.2 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests — Tests for description, crystallization point, assay and impurity [Sl No. (ii) and (iv), Table 1] shall be conducted on each to the individual samples.

4.3 Criteria for Conformity — The lot shall be declared as conforming to the requirements of description, crystallizing point, assay and impurities if each of the test results as obtained in **4.2** satisfy the corresponding requirements given in **2.1** and Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in IS : 5299-1969* and Appendix A as indicated in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†), shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHOD OF TEST FOR 4-TOLUIDINE AND 2-NITRO-4-TOLUIDINE IN 3-NITRO-4-AMINOTOLUENE

A-0. GENERAL

A-0.1 The expected impurities in 3-nitro-4-toluidine are 2-nitro-4-toluidine and 4-toluidine are estimated by ascending thin layer chromatographic method.

A-1. APPARATUS

A-1.1 Thin Layer Chromatographic Glass Plates — 20 × 20 cm.

A-1.2 Micropipette — of 10 μl capacity.

A-1.3 Chromatographic Chamber — 22 × 10 × 22 cm.

A-1.4 Adjustable Thin Plate Applicator

A-1.5 Chromatographic Spray Bottle

A-1.6 Weighing Bottle

A-2. REAGENTS

A-2.1 Sample Solution — 1 percent solution in ethanol.

A-2.2 Developing Solvent — Benzene.

A-2.3 Sodium Nitrite and Hydrochloric Acid — *N* $NaNO_2$ and *N* HCl solution (1 : 2) (*v/v*).

A-2.4 Coupling Reagent — Dissolve 0·1/g of 1 *N* naphthylethylene diamine dihydrochloride in a mixture of 50 ml of methanol and 50 ml of 10 percent hydrochloric acid (*v/v*).

A-2.5 Silica Gel, G — Neutral.

A-2.6 Reference Solution of 2-Nitro-4-Toluidine — (99·5 Percent Pure) — 0·005 percent solution in ethanol.

A-2.7 Reference Solution of 4-Toluidine — 0·005 percent solution in ethanol.

A-3. PROCEDURE

A-3.1 Preparation of Chromatographic Plates — Mix 50 g of neutral silica gel with about 90 ml of water for three minutes. Then by means of the applicator, coat the plate with the silica gel layer of thickness not exceeding 0·25 millimetre and place it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

A-3.2 Preparation of Sample Solution — Homogenize the sample for a few minutes, and weigh by means of a weighing bottle 1 0 g of the material and transfer to a 100-ml volumetric flask. Dissolve in ethanol and make up the solution to the mark by adding additional quantity of ethanol.

A-3.3 Chromatographic Separation — Spot 10 µl of the sample solution (**A-3.2**) and 10 µl of reference solutions (**A-2.6** and **A-2.7**) at a distance of 3 cm from the bottom edge. After complete evaporation of the solvent, place the plate in the chromatographic chamber and allow the developing solvent (**A-2.2**) in the chamber to migrate up to 13 cm. Remove the plate and let it dry in air at least 30 minutes. Dry the plate completely. Diazotize in nitrous fumes (**A-2.3**), and couple with coupling reagent (**A-2.4**) by spraying. The separated spots characteristic of constituents may be identified by their colour as under:

Content	*Zone*	*Rf Value*	*Colour*
4-Toluidine	I	0·26	Blue
2-Nitro-4-toluidine	II	0·3	Violet
3-Nitro-4-toluidine	III	0·4	Dark Violet

A-4. REPPORTING

A-4.1 Report impurity as that which is nearest in intensity to the reference sample.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s(s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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